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Quarterly Summary Report No. 4

ELASTOMERIC GASKET MATERIALS DEVELOPMENT FOR CRYOGENIC APPLICATIONS

Contract No. NAS 8-5053
Control No. TP 3-85370 (IF) CPB 02-1191-63

Jay W. Feldmann

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1 August 1963 through 31 October 1963

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NARMCO RESEARCH & DEVELOPMENT
A Division of Telecomputing Corporation
San Diego, California 92123

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National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Huntsville, Alabama

FOREWORD

This report, covering the period 1 August 1963 through 31 October 1963, was prepared by Narmco Research & Development, a Division of Telecomputing Corporation, San Diego, California, under Contract No. 8-5053, Control No. TP 3-85370 (1F) CPB 02-1191-63, entitled "Elastomeric Gasket Materials Development for Cryogenic Applications," for the George C. Marshall Space Flight Center of the National Aeronautics and Space Administration. The work was administered under the direction of the Propulsion and Vehicle Engineering Division, Engineering Materials Branch, George C. Marshall Space Flight Center, with Mr. J. E. Curry (R-P&VE-MNP) acting as Project Manager.

Prepared by: Jay W. Feldmann

Jay W. Feldmann
Senior Research Engineer

Approved by: Frank Wilson

Frank Wilson
Program Manager

B. L. Duft

B. L. Duft, Manager
Engineering Research Department

ABSTRACT

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A general review of the total program to date is presented. By comparing the work that has been performed and the results that have been obtained with the goals and objectives set forth in the contract, the areas requiring additional study are determined and future work planned. Author

TABLE OF CONTENTS

I. INTRODUCTION

The primary objective of this program is the development of new flat gasket materials suitable for use in the liquid oxygen (LOX) system of the Saturn launch vehicle. A secondary goal of this program shall be the evaluation of the same or appropriately modified gasket materials for liquid hydrogen.

II. DISCUSSION

A. Work Performed During Reporting Period

Fabrication of laminates for the second Greco-Latin square was completed during the last reporting period. These laminates were tested, and the energy absorbed by each is plotted in Figure 1. The following results were drawn from this second square analysis: the relative importance of the parameters is fabric weave, fabric style, and laminating temperature, respectively. Indicated optimum values were Crowfoot weave, 225-yard type, and 700°F, respectively.

Phase II, Metal Reinforced Laminates, was started. Laminates using reinforcement of corrugated aluminum, stainless steel screening, and stainless steel knit were fabricated and tested. Testing indicated that the metal reinforced laminates possessed lower compressibility than the glass fabric laminates. The metal reinforcement does not allow the controlled saturation feature as does the glass fabric; thus it does not have built-in mechanical compressibility. From a compressibility standpoint, the performance of these laminates was about the same as the solid resin. However, because of the reinforcement, the cold flow, or creep, properties were much better for the laminates than for the solid resins. More study is required on this phase before any definite conclusions can be drawn.

Phase III, Encapsulation of Gaskets, was also started during this reporting period. Preliminary testing indicated that the optimum Teflon-glass gasket would be fabricated from glass layers partially saturated with TFE resin

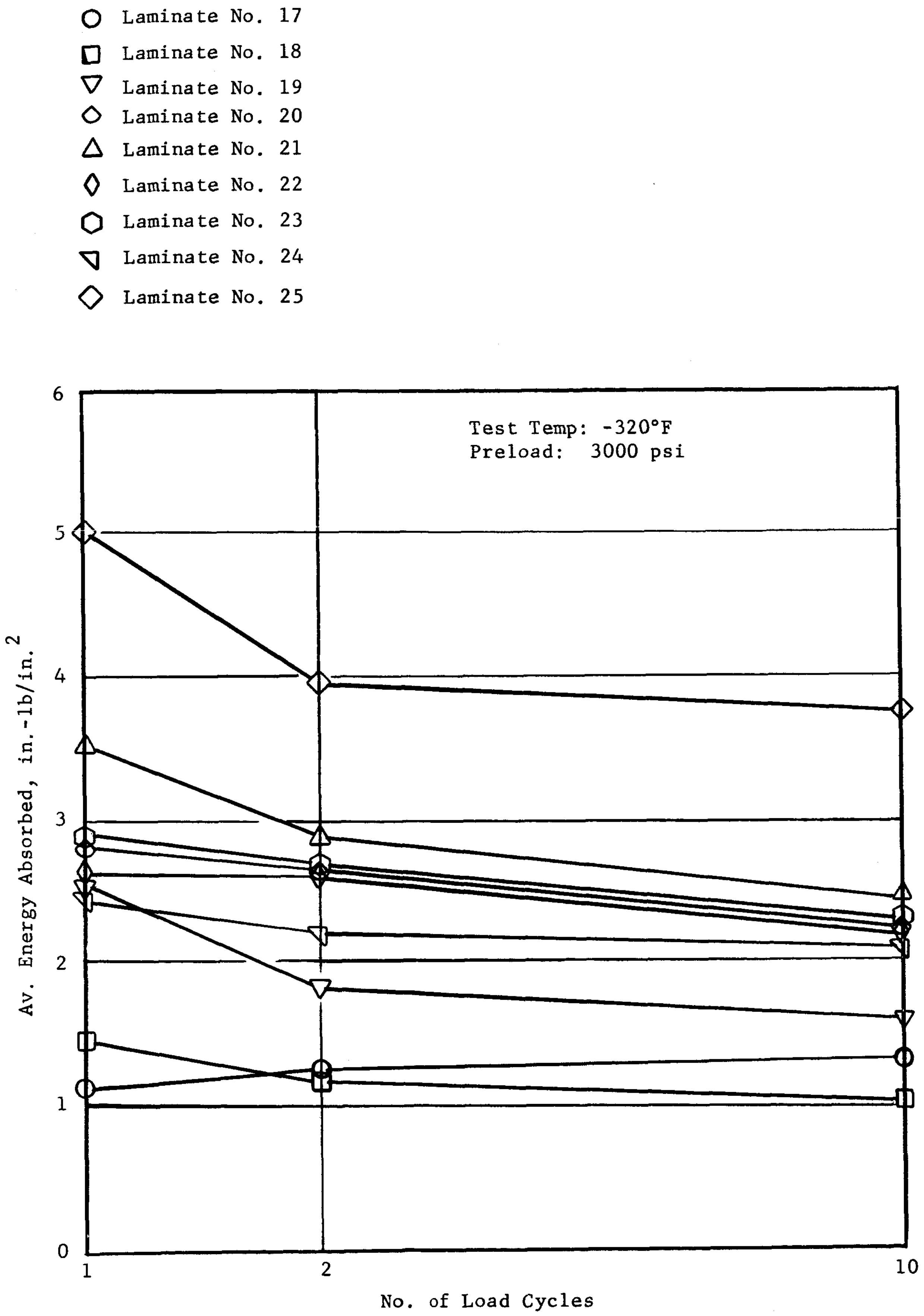

Figure 1. Energy Absorption vs. Load Cycle

and encapsulated with FEP resin. Since TFE resin flows at a higher temperature than FEP resin, the glass could be partially saturated with TFE at a high temperature, then encapsulated with FEP resin at a lower temperature. In this manner, the controlled saturation of the glass plies could be preserved, as well as the resultant compressibility. In the fabrication of the five large gaskets for NASA testing, major difficulties were encountered in bonding the encapsulation FEP resin to the TFE laminate. Because of the time limitation, it was necessary to use FEP resin for both the resin-glass laminates and the encapsulation material. It was realized that the encapsulation process would result in additional laminate resin flow and would decrease the compressibility of the gaskets. This, however, seemed to be the most reasonable approach considering the time limitation on the delivery of the five gaskets. Additional studies have resulted in the successful bonding of TFE and FEP resin. It now appears feasible to partially impregnate glass fabric with TFE and encapsulate with FEP without destroying the controlled resin saturation which results in the good compressibility of the gasket. It is considered that this combination will result in an optimum Teflon-glass laminate since the TFE-glass fabric appears to be the best laminate, from a compressibility standpoint, and the FEP with its superior flow and wetting properties provides the best encapsulation material. Therefore, the optimum combination would be a TFE resin laminate encapsulated with FEP.

Finally, specimens were cut from the laminates used to fabricate these five gaskets. Three separate tests are presently being conducted on each of these laminates: a leak test at room temperature, a leak test at cryogenic temperature, and a load-deflection test (over 10 cycles) at cryogenic temperature. A comparison of the results of these tests with those yet to be conducted will help predict how the optimum construction would have performed in the NASA test.

The laminating process used in the fabrication of the five gaskets is given below:

> The laminates were formed by alternating layers of 0.005-in. thick FEP Teflon sheet and glass cloth (Laminates A, B, and C used 181 cloth, while D and E used Crowfoot satin weave cloth). The outer layers of the sandwich have double layers of Teflon. The laminating temperature was 600°F, and the laminating pressure 50 psig.

Encapsulation was achieved by the following process:

> The gaskets were prepared by first cutting the rings from the laminate to the dimensions given on MSFC Drawing SK10-1433. Then, the inside diameter was opened by trimming away 0.05-in. (0.10-in. on the diameter). The rings were then placed one at a time in a mold of the final dimensions required by the drawing, the void was filled with FEP Teflon, and the laminating temperature and pressure given above were again attained.

B. Review of Total Program

This review of the total program is presented for the purpose of summarizing known results and conclusions in order to evaluate progress to the midpoint of the program extension, and to revise the work schedule so that the program objectives can be more efficiently attained.

1. Brief Chronological History

The dual requirements of cryogenic application and LOX compatibility reduced the field of possible gasket materials to one: fluorocarbon polymers. A set of requirements that the desired gasket should meet was proposed. One requirement, compressibility, was emphasized in all subsequent work.

A testing procedure called "load decay" was utilized to determine compressibility and compression set for various candidates in the fluorocarbon polymer family. Commercially available gasket materials were compared with materials proposed by Narmco. Various fillers in different resins were considered. The effect of the filler level on the compressibility was found to be quite large.

These tests demonstrated the cold flow, or creep, problem encountered with most of the materials. To restrict the flow of the resin, reinforcement in the form of glass fabric was found to be superior to the chopped fiberglass filler of any level. Therefore, a gasket material in the form of a laminate (alternate layers of resin and reinforcement) was conceived.

A series of tests ~~was~~ were performed whereby stress-strain curves were obtained for the various candidates over a 10-cycle loading. These tests allowed the determination of compressive moduli, which is an inverse measure of compressibility; i.e., higher modulus means lower compressibility, and vice-versa. In addition to offering a comparison of the various candidates, these tests showed how each candidate was affected by repeated loading (the modulus of the tenth cycle was always higher than that of earlier cycles, indicating an expected reduction in compressibility).

By comparing the amount of modulus change, information was obtained on the degree of constancy of the compressibility of the various materials, in addition to the absolute values for the materials. Finally, the moduli of all materials were higher (meaning lower compressibilities) at the cryogenic condition compared to room temperature condition. The fluorocarbon polymer laminate exhibited the least change in modulus with temperature. This feature is desirable, because in actual application on the Saturn, the gaskets will be bolted at room temperature. The cryogenic material will then be introduced. The material whose modulus and compressibility is least affected by this temperature change should maintain better seals without changing any bolting loads.

The improved laminate compressibility performance mentioned above is primarily due to a special laminating procedure developed by Narmco. The "sandwich" is prepared by stacking alternate layers of resin sheet and glass fabric until the desired thickness is obtained. The laminating temperature and pressure are controlled so that resin flow is sufficient for bonding the plies together, but not enough to completely saturate the glass fibers with resin. The unsaturated laminate has a mechanical compressibility built into it due to the unsaturated fabric. This mechanical compressibility is not affected by temperature. Hence, the unsaturated laminate possesses a modulus which is relatively unaffected by lowered temperature. In addition, it also has the lowest modulus (highest compressibility) at the cryogenic conditions, again due to the built-in mechanical compressibility.

Tests were performed so that hysteresis loops (stress vs. deflection over complete cycles) could be plotted for various materials, demonstrating differences in compression set and flow. Again, the Teflon glass fabric laminate performed the best.

Leak tests were performed in order to determine the minimum flange pressure required to retain an internal pressure. This allows the calculation of the ASME "m" factor for each material ("m" is the ratio of minimum flange pressure for no leak to internal pressure). Unfortunately, the various materials did not perform differently enough in this test to provide any clear distinctions. Therefore, the "flange deflection-to-leak" test was devised to find a better means of comparing the various candidates. This test consisted of measuring the amount of deflection a flange could undergo before the gasket leaked. While the Teflon glass fabric laminates performed no better than the average candidate in this test at room temperature conditions, they were by far the best at cryogenic conditions, and the differences between room temperature and cryogenic values were the smallest for these laminates.

A difficulty developed during testing, which was diagnosed as being caused by the protrusions on the gasket face caused by the shearing manner in which the gaskets were cut from the laminates: at the cryogenic condition, these protrusions offered resistance to the load and thereby influenced the test results. The remedy was to preload the gasket at room temperature, which flattened the protrusions and thereby created a truly flat gasket. After removing the load, the cryogenic condition could be imposed, and any desired testing could proceed. This procedure was later abandoned because the gaskets were machined, eliminating the protrusions and the need for this initial room temperature preload.

Having decided from the above tests that the glass fabric reinforced laminate appeared to be the best candidate, a program schedule was prepared with Phase I being the optimization study for this laminate. To perform this optimization, a statistical approach was taken using the Greco-Latin square technique. However, the Greco-Latin square allows only four variables. Since it was believed that a total of eight variables affected the laminate, four of the original eight quantities were taken as being constant, and a square was constructed with the other four variables having ranges of values. Unfortunately, some of the combinations of temperatures and pressures that were assigned to particular laminates were outside of the range of values necessary for flow of the various resins, or were so high that the resin burned, making lamination at the designated conditions impossible. The laminating conditions were subsequently modified to more realistic values which allowed lamination, but these changes destroyed the pattern of the Greco-Latin square and prevented its use as a statistical tool. However, the data that were obtained from the testing of the laminates were still of considerable value.

With the introduction of TFE resins, the stress-strain curves (load-deflection tests) no longer had straight line shapes but were curved, because the laminates did not deform linearly with load. Since it was considered desirable to continue comparisons on compressibility (which previously were based on compressive moduli), the moduli of the various laminates could not be determined as before, when they were simply the slope of the stress-strain curve: now there was no one slope. The problem was solved by the introduction of a new measure of comparison: the energy absorbed by the gasket during the loading cycle. This energy is the area under the stress-strain curve and was obtained by taking the average of three polar planimeter readings of the area. This quantity could have been converted into an equivalent modulus by determining the required base of a hypothetical triangle having the same area and then the slope of the hypothetical hypotenuse. However, it was felt that direct comparisons of the energies themselves would be just as meaningful, so this conversion was not attempted, since the higher the energy absorbed value, the more compressible the candidate.

The results of the tests run on the first group of laminates showed that Teflon TFE laminates had the highest energy absorption, followed by the Teflon FEP. Also, the lower laminating pressures resulted in better energy absorption properties.

Based on these tentative results, a second Greco-Latin square was constructed which did not maintain the same quantities as constants and variables that the first square did. This time, only three variables were considered: laminating temperature, fabric style, and weave. Tests were made on the various laminates and the results fed back into the Greco-Latin square. The results showed that the most important parameter was fabric weave, followed by fabric style, and laminating temperature. Of the parameter values considered, the indicated optimums are Crowfoot weave, 225-yard type, and 700°F, respectively.

Phase II, the study of metal reinforced laminates, was recently initiated. Laminates using different reinforcements were fabricated and tested, the results of which showed a lower compressibility of metal reinforced laminates then exhibited by the more optimum glass fabric laminates. (See Section A of this report for more details on this phase.) Work is continuing in this area.

Phase III, the encapsulation studies, has yielded some results. It should be recalled that the need for encapsulation arose because of the process used in lamination of the glass fabric candidates, where incomplete fiber wetting is desired in order to improve compressibility of the laminate. However, when gaskets are cut from the laminate, the inside and outside diameter surfaces expose the center of the laminate, where the resin has not saturated the fabric. If placed in a flanged joint, the material contained in the piping could leak through this section of the gasket. To prevent this type of gasket leakage, it is required that this possible path be blocked. This can be accomplished by completely covering the entire gasket with resin or simply by applying a band of resin to the inner diameter surface. Both approaches have been taken. (See Section A of this report for additional information.)

The encapsulated laminate desired (TFE resin-glass fabric laminate encapsulated with FEP) now appears to be within reach. Only optimization of the process remains. It should be pointed out that Phase III on encapsulation has only been concerned with the candidates from Phase I, Glass Reinforced Laminates. The metal reinforced laminates (Phase II) have not required encapsulation because they have not had a dry center for possible leakage. However, it is quite conceivable that some laminates of this type may be developed in Phase II, thereby requiring encapsulation and additional work in Phase III.

Phase IV, concerned with design criteria, is just being started.

2. Results and Conclusions to Date

PHASE I - A laminate of TFE Teflon and glass fabric has been found to provide the best gasket material in the nonmetallic reinforced group.

PHASE II - From Phase I, the best resin has been found to be Teflon TFE. Studies are still underway to determine the type of metal reinforcement that produces the optimum laminate.

PHASE III - Encapsulation has been required only for the candidates from Phase I (glass fabric reinforcement), and the optimum candidate (using TFE Teflon resin) should be encapsulated with FEP Teflon for the best result.

PHASE IV - The work to date has consisted of a brief literature survey. No results or conclusions have been reached.

III. ANTICIPATED WORK

PHASE I - Having determined the resin to be used, attention now will be directed to the possibility of using multi-axially woven fabrics for multi-directional strength increase. Also, studies will be made on the effects of varying the thicknesses of the plies of resin and/or fabric reinforcement so that the "best" laminate, with the minimum number of plies, can be determined. Also, the potentials of glass-wound reinforcements will be explored. Finally, the test results of the specimens from the laminates used to prepare the five NASA large diameter gaskets will be obtained, reduced, and analyzed.

PHASE II - More analysis is needed concerning the designs already proposed, and, possibly, different constructions might be considered. Because the proportions of the gaskets sent to NASA (large diameter, narrow flange) differ considerably from the test specimens used to date (small diameter, wide flange), it is believed that metal reinforced gaskets would be stronger than the glass fabric laminate for this extreme gasket design since it could support more hoop stress and therefore be less likely to "blow out." Consequently, more attention will be directed to metal reinforced laminates.

PHASE III - The optimization of the encapsulation of the glass reinforced laminate remains; i.e., determining the best combination of bonding temperature and pressure. As mentioned earlier in this report, it is quite possible that some candidate will be developed from Phase II that will require encapsulation, also.

PHASE IV - The design and performance criteria study is now in process. It is anticipated that much help will be supplied by the quarterly reports of the General Engineering Laboratory's NASA Study "Design Criteria for Zero-Leakage Connectors for Launch Vehicles." It is requested that Narmco be supplied with the final report as soon as it is available.

PHASE V - This phase will cover the liquid hydrogen application for the best candidates from the LOX application studies. Since this is the secondary goal of the program, it is not planned to initiate this work until the LOX gasket study is sufficiently complete for data comparison. Therefore, it is now scheduled to begin in January 1964 and will continue as funds and time allow.

IV. PROGRAM SCHEDULE

Based on the anticipated work outlined above, a revised schedule is proposed (see Figure 2). Note the extension of studies of the first two phases, which will now run until the end of the year. It is felt that further work is required in these areas. It is planned that the first three phases should be completed at about the same time (1 January 1964).

It is anticipated that the 2-month period for Phase V will be sufficient to obtain meaningful results for the liquid hydrogen phase of this program, and that the extension of the other phases will not result in additional funding or time requirements.

A total of 892 man-hours was expended during this reporting period. A total of 1451 man-hours has been expended during the current contract period.

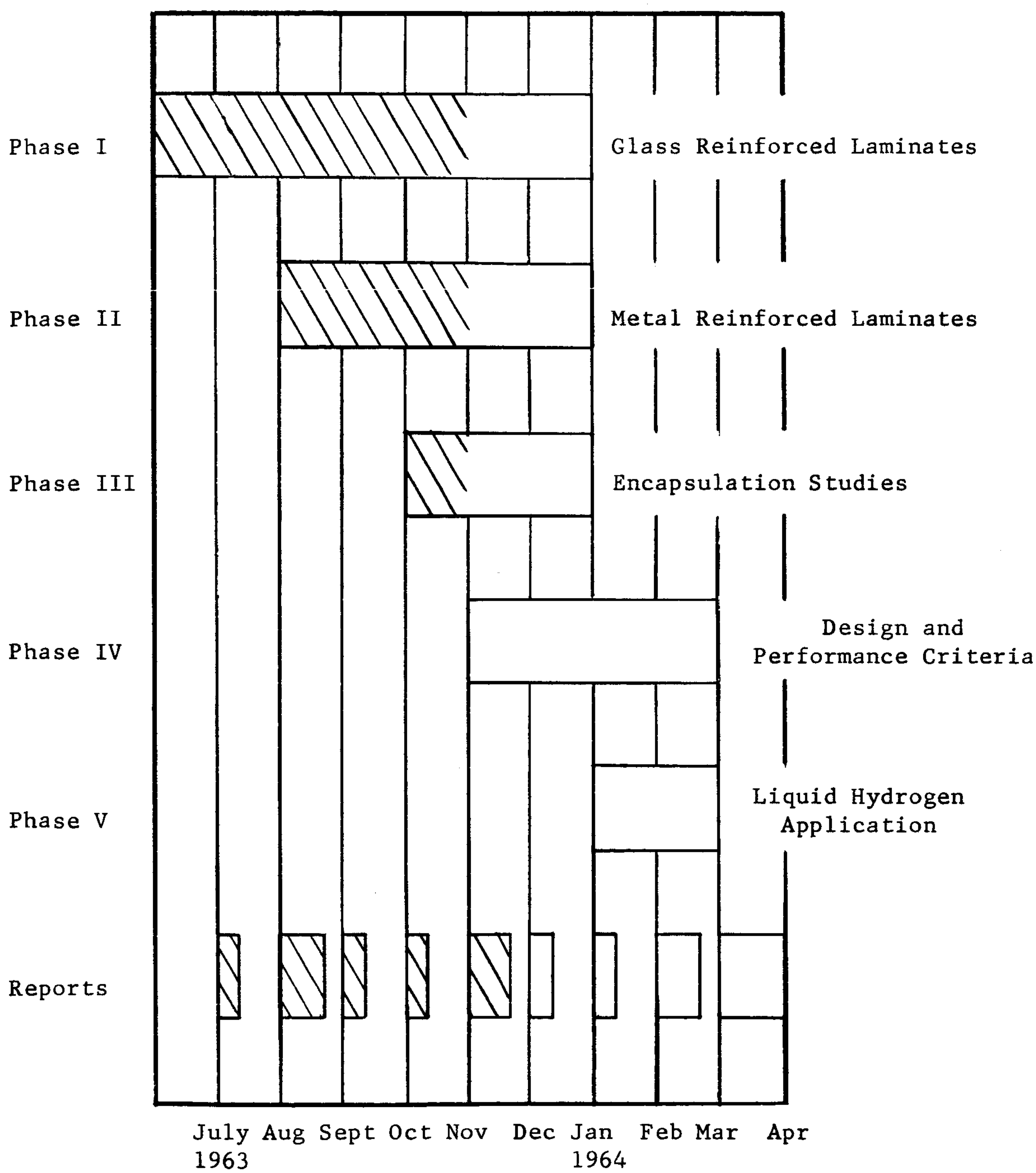

Figure 2. Program Schedule

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